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<p>(54) Title: ROCKET ASSEMBLY ABLATIVE MATERIALS FORMED FROM SOLVENT-SPUN CELLULOSIC PRECURSORS, AND METHOD OF INSULATING OR THERMALLY PROTECTING A ROCKET ASSEMBLY WITH THE SAME</p>		
<p>(57) Abstract</p> <p>A rocket motor assembly is insulated or thermally protected with a rocket motor ablative material formed from a prepreg. The prepreg contains at least an impregnating resin matrix and, as a precursor prior to carbonization, either carded and yarn-spun solvent-spun staple cellulosic fibers, solvent-spun cellulosic filaments, or a combination thereof. When patterned and carbonized, the rocket motor ablative material can be lined or otherwise placed into a rocket motor assembly, such as between the solid propellant and case, in the bulk area of the exit nozzle liner, or at susceptible portions of a re-entry vehicle, such as the nose cone.</p>		

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**ROCKET ASSEMBLY ABLATIVE MATERIALS FORMED
FROM SOLVENT-SPUN CELLULOSIC PRECURSORS,
AND METHOD OF INSULATING OR THERMALLY
PROTECTING A ROCKET ASSEMBLY WITH THE SAME**

5

10 **BACKGROUND OF THE INVENTION**

1. **Field of the Invention**

 This invention relates to rocket motor ablative materials, especially
resin-filled carbon fiber and carbon/carbon ablative materials, and a method
of making the ablative materials. In particular this invention relates to
15 carbon ablative materials having a reinforcement component formed from,
as a precursor prior to carbonization, carded and yarn-spun solvent-spun
cellulosic fibers and/or solvent-spun filaments. This invention also relates
to rocket motor assemblies including the carbon ablative materials.

2. **Description of the Related Art**

20 It is generally accepted current industry practice to prepare
insulation for solid propellant rocket motors from a polymeric base
composite importantly including a carbon cloth. The composite is generally
composed of the carbon cloth as a woven reinforcement structure
impregnated with a suitable resin matrix. The resin matrix is commonly a
25 phenolic resin, although other resin matrices can be used. For making the
woven reinforcement structure, current industry practice is to select a
continuous filament of non-solvent-spun viscose rayon as a precursor
material (hereinafter referred to as continuous filament viscose rayon). The
continuous filament viscose rayon, which is especially formulated for
30 ablative applications, is woven, wound, or otherwise manipulated into its

desired configuration and then carbonized to form a carbon structure exhibiting superior ablation characteristics and excellent physical properties and processability.

Continuous filament viscose rayon precursor has been established as a standard in the rocket motor industry for making carbon reinforced structures of carbon and carbon/carbon ablative materials due to its superior ablation characteristics, excellent physical and thermal properties, and high processability. One of the excellent physical properties possessed by composites formed from continuous filament viscose rayon precursor is a cured composite high warp strength of about 144.8 MPa (about 21,000 lbs/in²) at ambient temperature (about 21°C or 70°F), as measured subsequent to carbonization and impregnation of the precursor. Warp strength reflects the tolerance of the filament to opposing forces acting along the warp (or longitudinal) filament axis.

However, a major drawback associated with the use of cured composites comprising wrapped layers of continuous filament viscose rayon, such as found within the bulk areas of much rocket nozzle insulation, is the relative low across-ply tensile strength possessed by the carbonized continuous filament viscose rayon at operating temperatures experienced within the bulk ablative material (as opposed to the exhaust gas surface) during firing of a rocket motor. Such firing temperatures within the bulk ablative material generally can rise to about 400°C (or 750°F). Specifically, cured composites comprising wrapped layers of carbonized continuous filament viscose rayon have across-ply tensile strengths on the order of about 2.07 MPa (about 300 lbs/in²). As referred to herein, across-ply tensile strength is the amount of load, perpendicular to the filament axes, which two overlapping layers of filaments can withstand prior to slippage.

Another significant drawback associated with continuous filament viscose rayon that has recently drawn significant attention involves the availability of this particular type of continuous filament. Over the past few years, the only manufacturer producing sufficient quantities of non-solvent-spun continuous filament viscose rayon to meet industry demands is North American Rayon Corp. (NARC) of Elizabethton, Tennessee. The capability of the industry to produce ablative liners and other thermal insulation based on continuous filament viscose rayon has been jeopardized, however, due to the cessation of continuous filament viscose fiber production by NARC. There is therefore a need in this industry, previously not satisfied, to find an effective alternate source or a replacement candidate for the above-described standard thermal insulation formed from continuous filament viscose rayon precursor.

The requirements that a replacement candidate must satisfy in order to be acceptable and functionally effective are well known to be quite severe due to the extreme conditions to which the insulation is exposed. These conditions not only include exceedingly high temperatures but also severe ablative effects from the hot particles (as well as gases) that traverse and exit the rocket motor interior, or over the outer surface of re-entry vehicle insulators. Unless the insulation will withstand such conditions, catastrophic failure may (and has) occurred.

Accordingly, any replacement insulation should exhibit comparable temperature resistant and ablation characteristics and rheological and physical properties at least equivalent to those of continuous rayon viscose filament, yet should not otherwise significantly alter the manufacturing process employed for the production of the thermal insulation. Additionally, due to the large and growing quantities of solid propellant rocket motor insulation required by the industry, any such replacement

reinforcement precursor candidate should be abundantly available now and into the foreseeable future.

An alternative carbon precursor that has been proposed for ablative applications is continuous filament polyacrylonitrile (PAN). PAN
5 continuous filaments disadvantageously possess higher densities than cellulosic materials (1.8 g/cm^3 for PAN, compared to 1.48 g/cm^3 for cellulosic filaments) and higher thermal conductivities than cellulosic materials. Thus, in order to provide a comparable insulation performance to rayon filaments, rocket motor nozzle insulation or re-entry vehicle
10 insulation formed from PAN filament must have a greater thickness and weight than a comparable-performing insulation formed from cellulosic materials. The replacement material must meet the ablation limits for protection of the casing (when used as an internal casing insulation) throughout the propellant burn without adding undue weight to the motor.

15 Accordingly, the search for a functionally satisfactory precursor for making the reinforcement structure of a composite material requires discovery and implementation of an extraordinarily complex combination of characteristics. The criticality of the material selection is further demonstrated by the severity and magnitude of the risk of failure. Most
20 insulation is of necessity "man-rated" in the sense that a catastrophic failure can result in the loss of human life -- whether the rocket motor is used as a booster for launch of a rocket or is carried tactically underneath the wing of an attack aircraft. The monetary cost of failure in satellite launches is well-publicized and can run into the hundreds of millions of dollars.

25 Therefore, one of the most difficult tasks in the solid propellant rocket motor industry is the development of a suitable, acceptable insulation that will meet and pass a large number of test criteria to lead to its acceptability.

Furthermore, any replacement precursor should not be susceptible to obsolescence issues nor discontinuance in future supply thereof.

SUMMARY OF THE INVENTION

It is, therefore, an object of this invention to address a crucial need
5 in the industry to reformulate the ablative liners and thermal liners of rocket
motors by finding a suitable replacement precursor for making carbon-
based reinforcement structures. As referred to above, suitable replacement
means a precursor material that can be substituted for continuous filament
viscose rayon without requiring significant amounts of modification to the
10 impregnating resin composition, component design, and manufacturing
process steps and, when carbonized, possess equal or superior properties, in
particular overall strength, as the those possessed by the continuous
filament viscose rayon standard.

In accordance with the principles of this invention, these and other
15 objects of the invention are attained by the provision of a rocket motor
ablative material (*e.g.*, an insulation liner or the like) formed from, as a
precursor of the carbon reinforcement structure, yarn comprising either
carded and yarn-spun solvent-spun cellulosic (*e.g.*, rayon) fibers, solvent-
spun cellulosic filaments, or a combination thereof. The inventors
20 discovered that solvent-spun cellulosic fibers and filaments are capable of
being processed, such as by spinning, into yarns which, upon patterning
(*e.g.*, weaving in any weave style or winding) and subsequent
carbonization, can serve as a reinforcement of prepreps and can be
processed into an insulation liner under conditions comparable to those of
25 continuous filament viscose rayon.

The inventors also discovered that when carded and yarn-spun
solvent-spun staple cellulosic fibers possessing certain dimensional

characteristics are selected, the resulting yarn possesses excellent mechanical strength for rocket motor applications, yet does not release unacceptable levels of fiber fly -- *i.e.*, short, waste fibers -- into the air in textile processing operations such as carding, yarn-spinning, and weaving.

- 5 The former discovery, in particular, was especially surprising because yarns prepared from cellulosic fibers were expected to possess and do possess significantly lower warp strengths than yarns produced from continuous filament viscose rayon. However, the inventors found that the lower warp strengths of the yarns prepared from cellulosic fibers are compensated for
- 10 by the far superior across-ply tensile strength that yarns prepared from cellulosic fibers exhibit over continuous filament viscose rayon.

This invention is also directed to a rocket motor assembly comprising ablative materials which comprise reinforcing structures formed from, as a precursor material prior to carbonization, yarn comprising either

15 carded and yarn-spun solvent-spun cellulosic (or rayon) fibers, solvent-spun cellulosic filaments, or a combination thereof. This invention is further directed to a process for making a rocket motor assembly comprising the ablative materials including nozzle and re-entry vehicle components.

Other objects, aspects and advantages of the invention will be

20 apparent to those skilled in the art upon reading the specification and appended claims which, when taken in conjunction with the accompanying drawings, explain the principles of this invention.

BRIEF DESCRIPTION OF THE DRAWINGS

The accompanying drawings serve to elucidate the principles of this

25 invention. In such drawings:

FIG. 1 is a schematic cross-sectional view depicting the insulation of this invention interposed between a rocket motor casing and solid propellant;

FIG. 2 is a schematic cross-sectional view identifying some of the regions of a rocket motor assembly in which the insulation of this invention may be applied; and

FIG. 3 is a flow diagram of a process for making LYOCELL fibers.

DETAILED DESCRIPTION OF THE INVENTION

In accordance with the principles of this invention, the replacement precursor material for preparing carbon reinforcement structures of rocket motor ablative materials, including nozzle re-entry vehicle components, is yarn comprising either carded and yarn-spun solvent-spun cellulosic (or rayon) fibers, solvent-spun cellulosic filaments, or a combination thereof. As referred to herein and understood in the art, carded means fibers subjected to a process or passed through a machine designed to promote the at least partial separation and at least partial alignment of fibers. Carding encompasses techniques used in the production of both fine and coarse yarns. As referred to herein and understood in the art, yarn-spun means a yarn formed a combination of drawing or drafting and twisting of prepared fibers. Yarn-spinning as referred to herein is not intended to mean techniques consisting of the extrusion of continuous filaments, which techniques can be performed during solvent-spinning. As referred to herein, staple fibers are fibers having lengths suitable for yarn-spinning.

Various solvent-spun cellulosic fibers can be used in accordance with this invention. A representative example is of a solvent-spun cellulosic fibers are LYOCELLcellulosic fibers made by spinning with N-methylmorpholine-N-oxide.

The solvent-spun cellulosic fibers preferably have average fiber lengths in a range of from 38 mm to 225 mm, such as 100-150 mm. The solvent-spun cellulosic fibers, when processed into a yarn, and/or solvent spun into filaments preferably have an average denier per filament (dpf) in
5 a range of from 1.1 dpf to 3.0 dpf. One supplier of solvent-spun cellulosic fibers and filaments is Lenzing Fibers of Austria. The solvent-spun cellulosic materials provided from this commercial source generally have sodium and zinc levels of 90 ppm and 2 ppm, respectively, which are less than the 1300 ppm and 300 ppm of typical continuous filament viscose
10 rayon supplied by NARC. Solvent-spun cellulosic fibers made by solvent-spinning with N-methylmorpholine-N-oxide are commonly known as LYOCELL.

The cellulosic fibers and filaments are preferably untreated, meaning that they are free of any distinct metallic, metalloidal, or graphitic
15 coating, at least prior to (and preferably subsequent to) graphitization.

One of the advantageous features of this invention is that the yarn comprising the solvent-spun filaments and/or the carded and yarn-spun solvent-spun cellulosic fibers may be substituted for conventional continuous filament viscose rayon without significantly altering the ablative
20 material manufacturing process. The only substantial alteration in the manufacturing process, at least with respect to the processing of reinforcement structures from solvent-spun cellulosic fibers, resides in the differences between producing the yarn of this invention and producing conventional continuous filament viscose rayon. Generally, continuous
25 filament viscose rayon is produced by dissolving cellulose into a viscose spinning solution, and extruding the solution into a coagulating medium where the polymer is cellulose and is regenerated as a continuous filament. On the other hand, the yarn used in an embodiment of the present invention

is prepared from solvent-spun staple fibers, which are carded and yarn-spun by techniques well known in the industry into a tight, compact yarn from the staple fibers. It is understood that other processing techniques may also be used, such as combing and other steps well known and practiced in the art. Preferably, the yarn-spinning step is performed by either a worsted process or cotton-ring spinning process. The yarn-spinning process is advantageous to keep yarn hairiness to a minimum. By way of example, the yarn may have a weight comparable to the weight of standard yarns presently used for carbon ablative materials, i.e., about 1650 denier. This may be accomplished with staple fibers by producing a yarn that is approximately 4.8 English worsted count (Nw), and two-plying the yarn to obtain the 1650 denier configuration. Suitable amounts of twist attained by spinning can be, for example, 2-12 360° turns per inch, more preferably 10-12 360° turns per inch.

The yarns are then subject to one or more patterning techniques, including, by way of example, weaving, winding, and plying, into a desired structure. The structure is then carbonized to form the reinforcement of the ablative material. In this regard, the structuring of the yarns into the desired configurations can be performed in the same manner as that for conventional continuous filament viscose rayon. Carbonization can take place, by way of example, at a temperature of at least 1250°C, preferably at least 1350°C. The carbonized reinforcement structure is then impregnated with an acceptable resin, such as a phenolic resin. A representative phenolic resin is SC1008, available from Borden Chemical of Louisville, Kentucky.

The inventive ablative and insulation materials can be applied to various parts of a rocket assembly, preferably as multi-layered structures. For example, the ablative and insulation materials can be used as a chamber internal insulation liner, as shown in FIG. 1. Referring to FIG. 1, the

insulation 10, when in a cured state, is disposed on the interior surface of the rocket motor case 12. Typically, a liner 14 is interposed between the case 12 and the insulation 10. The insulation 10 and liner 14 serve to protect the case from the extreme conditions produced by the burning propellant 16. Methods for loading a rocket motor case 12 with an insulation 10, liner 14, and propellant 16 are known to those skilled in the art, and can be readily adapted within the skill of the art without undue experimentation to incorporate the insulation of this invention. Liner compositions and methods for applying liners into a rocket motor case are also well known in the art, as exemplified by U.S. Patent No. 5,767,221, the complete disclosure of which is incorporated herein by reference.

The ablative and insulation materials can also (or alternatively) be applied along the flow path through which the combustion products pass, such as shown by the shaded area 20 of the exit nozzle shown in FIG. 2.

The ablative performance and mechanical properties of the carbon cloth phenolic prepared from cellulosic fibers as a precursor to the reinforcement are comparable those of carbon cloth phenolics made from the aerospace-grade continuous filament viscose rayon in subscale test motors. For example, although carbonized yarns formed from solvent-spun cellulosic fibers exhibit a slightly lower warp strength than yarns formed from continuous filament viscose rayon (96.5 MPa (or 14,000 lbs/in²) compared to 144.8 MPa (or 21,000 lbs/in²)), carbonized yarns formed from solvent-spun cellulosic fibers have an across-ply strength (5.52 MPa or 800 lbs/in²) twice that of continuous filament viscose rayon (2.76 MPa or 400 lbs/in²) (at rocket firing temperatures). Although this invention is not currently intended to be limited by any theory, it is believed that the enhanced across-ply tensile strength of the inventive ablative material is attributable to the orientation of fibers being offset relative to the yarn axis.

As a result, the ends of fibers forming the yarn can entangle with the fibers of an adjacent layer of yarn, thereby increasing the shear strength between the layers of yarn.

This invention will now be described with reference to the following examples, which are neither exhaustive nor exclusive of the scope of this invention.

EXAMPLES

Example 1

3.0 dpf LYOCCELL staple fibers having an average length of about 51 mm were spun into yarns having an average denier of about 825 using a cotton ring spinning machine.

Example 2

3.0 dpf LYOCCELL staple fibers having an average length of about 100 mm were spun into a yarn having denier of about 825 a using a worsted wool spinning machine.

For each of Examples 1-2, the yarns were spun into heavy tow yarns each having a denier of about 825. This was accomplished by making a Ne (Number English) 6.4 spun yarn. By two-plying (twisting) the yarn into a Ne 3.2 yarn, a denier of 1650 was obtained. The resulting yarns were then woven into fabric in a square woven having a 5 harness satin configuration. The fabrics were then carbonized using the standard carbonization schedules used for ablative carbon fabric filament cellulosic fibers.

The carbonized fabric was impregnated with a phenolic resin, and in particular phenol formaldehyde resole resin. The prepreg material was 31.0-36.0 wt% phenolic resin, 13.0-17.5 wt% carbon black filler, and 46.5 to 56.0 wt% carbon fabric.

The following table lists the thermal and mechanical properties of various yarns, carbon cloths, and carbon cloth phenolic ablative materials tested to compare staple cellulosic precursors with current filament rayon precursors.

5

Table 1

Properties of Filament and Solvent-Spun Staple Cellulosic Fibers in Carbon Cloth Phenolic Ablatives			
Yarn Properties	Units	Filament Rayon	Staple LYOCCELL (Example 1)
Yarn Denier	g/9 KM	1650	1650
Yarn Plies	Ply	1	2
Fibers per Yarn	-	720	550
Denier per Filament	Dpf	2.3	3.0
Woven Fabric Properties			
Fabric Width	cm (inches)	152 (60)	152 (60)
Area Weight	g/m ² (oz/yd ²)	576 (17.0)	576 (17.0)
Weave Pattern	-	8 harness satin	5 harness satin
Carbon Fabric Properties			
Fabric Width	cm (inches)	109 (43)	117 (46)
Area Weight	g/m ² (oz/yd ²)	271 (8.0)	271 (8.0)
Carbon Content	%	97.7	95.7
Prepreg Properties			
Carbon Content	%	50.6	45.6
Resin Content	%	34.2	38.4
Filler Content	%	15.2	17.0
Cured Composite and Ablative Properties			
Across Ply Tensile (@ 21°C or 70°F)	MPa (psi)	26.5 (3837)	32.2 (4665)

Across Ply Tensile (@ 399°C or 750°F)	MPa (psi)	2.12 (307)	5.21 (756)
Interlaminar Shear Strength	MPa (psi)	39.7 (5760)	50.1 (7267)
Nozzle Erosion Rate*	µm/sec (mils/s)	171 (6.74)	173 (6.82)
Total Heat Effected Depth*	mm (inches)	14.1 (0.556)	13.1 (0.516)
* Based upon solid fuel rocket test motor firing of 35 seconds at 900 psi.			

As shown in the above Table, the carbon cloth phenolic ablatives formed from staple LYOCELL fibers in accordance with this present invention exhibited much higher across ply tensile strengths than the conventional continuous filament viscose rayon precursor at room temperature of 21°C and operating temperatures of 399°C.

The foregoing detailed description of the preferred embodiments of the invention has been provided for the purposes of illustration and description. It is not intended to be exhaustive or exclusive in its description of the precise embodiments disclosed. The embodiments were chosen and described in order to best explain the principles of the invention and its practical application, thereby enabling others skilled in the art to understand the invention for various embodiments and with various modifications covered within the spirit and scope of the appended claims.

WE CLAIM:

1. A method for insulating or thermally protecting a rocket motor assembly comprising a rocket motor casing, a solid propellant, and a nozzle assembly, said process comprising (a) forming a rocket motor
5 ablative material from a prepreg comprising at least one impregnating resin matrix and, as a precursor prior to carbonization, either carded and yarn-spun solvent-spun staple cellulosic fibers, solvent-spun cellulosic filaments, or a combination thereof and (b) insulating or lining a portion of the rocket motor assembly with the rocket motor ablative material.
- 10 2. The process of claim 1, wherein the ablative material comprises the solvent-spun staple cellulosic fibers.
3. The process of claim 2, the solvent-spun staple cellulosic fibers have an average length in a range of from 38 mm to 225 mm, and are spun into yarn having a denier per fiber in a range of from 1.1 dpf to 6.0
15 dpf.
4. The process of claim 1, wherein the precursor is made by solvent spinning with N-methylmorpholine-N-oxide.
5. The process of claim 1, wherein the solvent-spun staple cellulosic fibers are untreated.
- 20 6. The process of claim 1, wherein said insulating or lining step comprises applying the rocket motor ablative material between the solid propellant and the casing surrounding the solid propellant.
7. The process of claim 1, wherein said insulating or lining step comprises applying the ablative material as a bulk ablative material of an
25 exit nozzle liner.

8. The process of claim 1, wherein said insulating or lining step comprising applying the ablative material as a bulk ablative material of a re-entry vehicle nose cone.

9. The process of claim 1, further comprising carbonizing the prepreg at at least 1350°C.

10. The process of claim 1, wherein the prepreg comprises 31.0-36.0 wt% phenolic resin, 13.0-17.5 wt% carbon black filler, and 46.5 to 56.0 wt% carbon fabric.

11. A rocket motor ablative material comprising a prepreg, said prepreg comprising at least one carbon-based reinforcement structure impregnated with at least one resin, said reinforcement structure being formed from, as a precursor prior to carbonization, either carded and yarn-spun solvent-spun staple cellulosic fibers, solvent-spun cellulosic filaments, or a combination thereof.

12. The rocket motor ablative material of claim 11, wherein the ablative material comprises the solvent-spun staple cellulosic fibers.

13. The rocket motor ablative material of claim 12, wherein the solvent-spun staple cellulosic fibers have an average length in a range of from 51 to 225 mm, and are spun into yarn having a denier per filament in a range of from 1.1 dpf to 6.0 dpf.

14. The rocket motor ablative material of claim 11, wherein the precursor is made by solvent spinning with N-methylmorpholene-N-oxide.

15. The rocket motor ablative material of claim 11, wherein the solvent-spun staple cellulosic fibers are untreated.

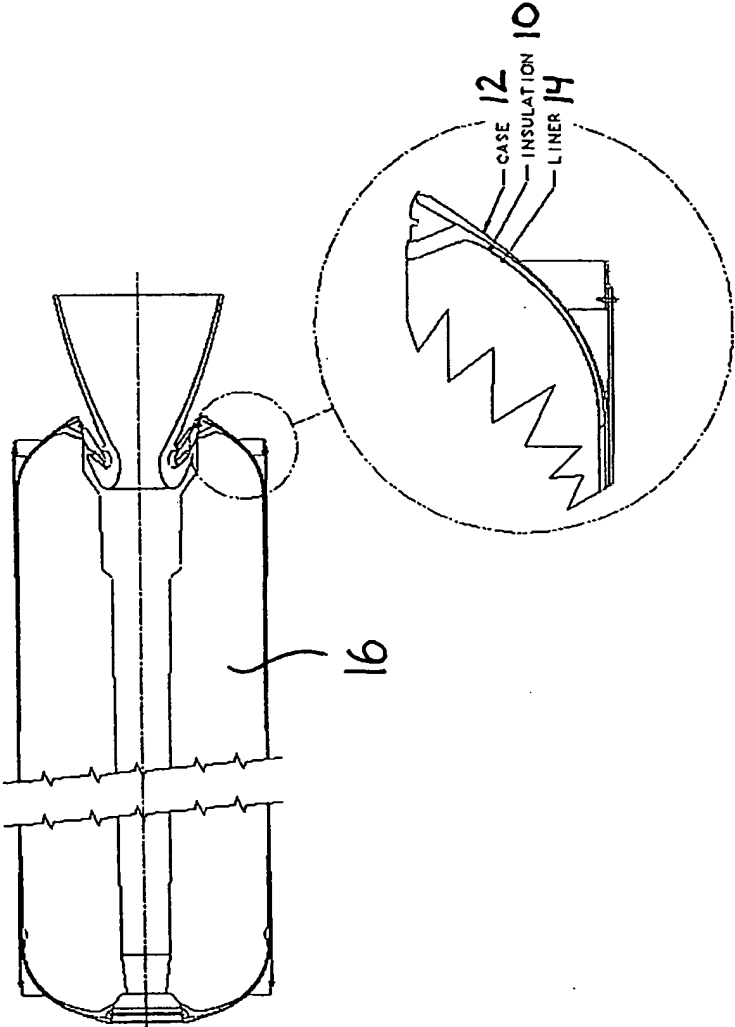
16. A rocket motor assembly comprising the ablative material of claim 11.

17. The rocket motor assembly of claim 16, wherein the ablative material is constructed and arranged as a bulk ablative material of an exit
5 nozzle liner.

18. The rocket motor assembly of claim 16, wherein the ablative material is constructed and arranged as a bulk ablative material of a re-entry vehicle nose cone.

19. The rocket motor assembly of claim 16, wherein the ablative
10 material is interposed between a solid propellant and casing of the rocket motor assembly.

FIG. 1



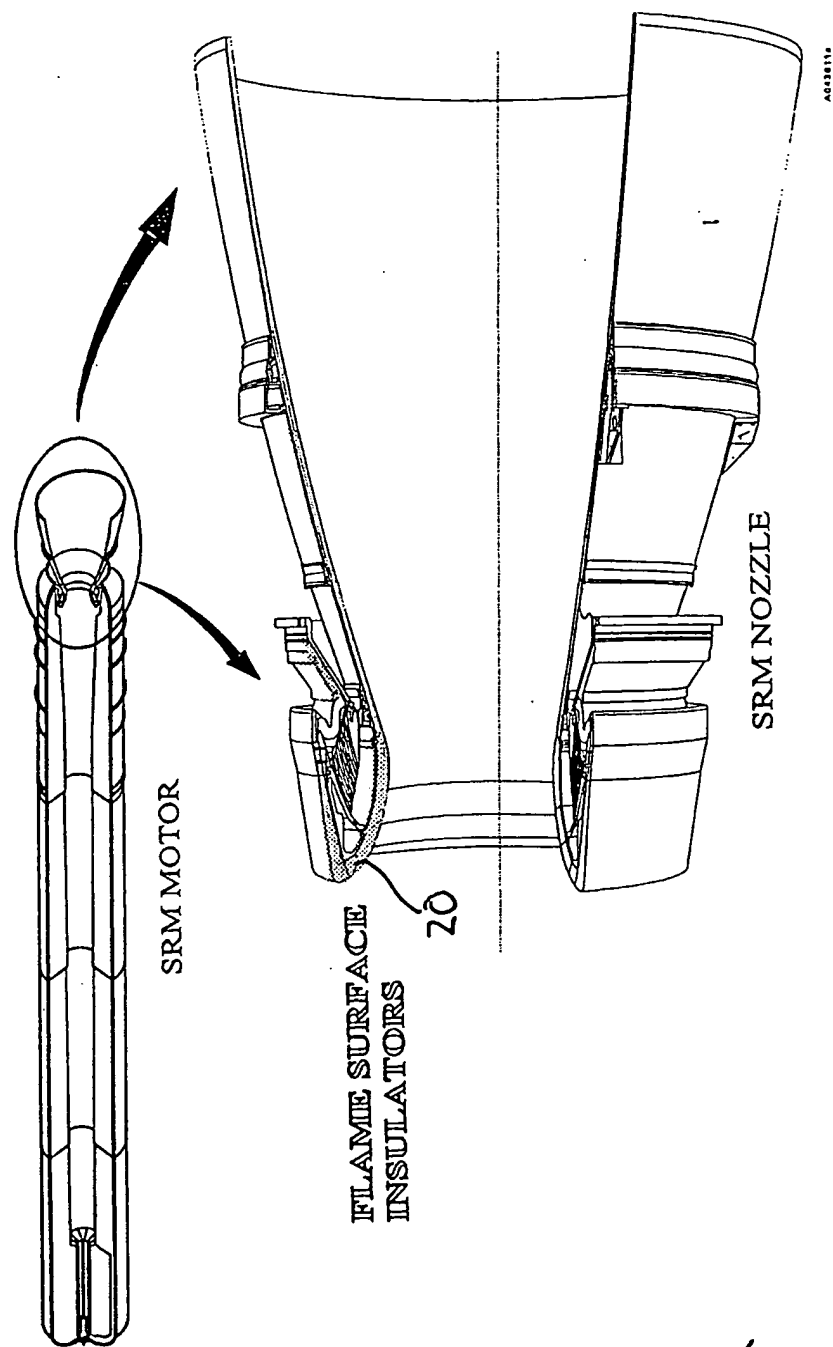


FIG. 2

FIG. 3

LYOCELL PROCESS

